# UNCLASSIFIED

AD 408 362

# DEFENSE DOCUMENTATION CENTER

FOR

SCIENTIFIC AND TECHNICAL INFORMATION

CAMERON STATION, ALEXANDRIA, VIRGINIA



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS

AñF 8 18è3 DDC

30 JANUARY 1963

UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

40836

C)

9

4083

CATALOGED BY

NOLTR 63-2

# HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS (U)

Summary Report: 30 June 1961 - 30 June 1962

Prepared by: F. J. Koubek

ABSTRACT: Oxy-acetylene torch test studies on a series of experimental phenolic resins are described along with those conducted on a number of commercial ablation type materials. A phenolic-polyamide copolymer was found to be the most effective for insulation effectiveness. The potential of silicone rubber as the matrix for an effective elastomeric insulator was demonstrated.

A thermal insulation study program, designed to gain an experimentally supported understanding of ablation mechanisms, is described. The intent is to apply this information to improved methods of design and selection of insulation materials. The general plans are to measure effective ablative properties at selected environmental parameters. Efforts have been presently directed toward further refinement of the "alpha-rod" test technique and investigating the requirements for a plasma are driven environmental simulator. An are image furnace is under construction for radiation and special atmosphere studies.

The efforts of a Bureau of Naval Weapons working committee, chaired by the Naval Ordnance Laboratory, to arrive at a standard reproducible oxy-acetylene screening test are described. Results of a round-robin test program and committee studies to improve reproducibility are discussed. The revised test method is now being evaluated in an interlaboratory test program.

PUBLISHED MAY 1963

APPROVED BY: F. ROBERT BARNET
Non-Metallic Materials Division

CHEMISTRY RESEARCH DEPARTMENT U.S. NAVAL ORDNANCE LABORATORY WHITE OAK, SILVER SPRING, MARYLAND HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS

For the past five years, the high temperature materials program at the Naval Ordnance Laboratory has consisted of a continuing study of materials behavior in high thermal environments. Information gathered in these studies is intended to act as a guide in the improvement of materials for high temperature use, as well as to provide optimization for specific applications.

This report is a statement of progress for the period of 30 June 1961 -30 June 1962. The work was conducted under the High Temperature Rocket Materials Program, RMMP 23-054/212-1/F009-06-003 and Task NOL 554 and was accomplished by Messrs. F. J. Koubek, W. J. McLean, D. M. Caum and B. T. Hartmann.

Many of the materials discussed in this report were obtained from commercial sources. Their evaluation by the Laboratory in no way implies Navy endorsement for their high temperature usage. Neither is this consideration of a material by the Navy to be used for promotional purposes. There is no implication intended that other materials might not have performed as well as those selected for these studies.

> R. E. ODENING Captain, USN Commander

ALBERT LIGHTBODY

By direction

### CONTENTS

	CONTENTS	
INTRODUC	TION	Page 1
Backgr	NSERT MATERIALS	3 3 3
Genera Modifi Rigid Flexib Silico Erosio Compos Summar	ON MATERIALS  ded Phenolic Resins Insulation Materials le Insulation Materials one Rubbers on Resistant Materials dite Structures  y ssions	3345666778
Genera Alpha- Variab Arc Im	ON STUDIES  Rod Ablation Technique  le Environmental Simulator  uage Furnace  y and Conclusions	9 9 10 10
Genera First Second Third	ROUND TEST STANDARDIZATION  Committee Meeting  Committee Meeting  y and Conclusions	12 12 12 12 12 14
RECOMMEN	DATIONS	14
	TABLES	
Table	Title	Page
1 2	Oxy-Acetylene Panel Test Reference Data on Typical Materials Oxy-Acetylene Panel Test Data - Phenolic Resin	16
ے	Studies	17
3	Oxy-Acetylene Panel Test Data - Resin Content	
4	Studies	18
	Reinforcement Studies	19

### TABLES (Cont.)

Table	Title	Page
5	Oxy-Acetylene Panel Test Data - Commercial Insulation Materials (Rigid Type)	20
6	Oxy-Acetylene Panel Test Data - Commercial Insulation Materials (Flexible Type)	
7 8	Oxy-Acetylene Panel Test Data - Silicone Rubbers Oxy-Acetylene Panel Test Data - Commercial Erosion	
	Resistant Materials	23
9 <b>1</b> 0	Oxy-Acetylene Panel Test Data - Composite Structures  Analyses of Round-Robin Data (Oxy-Acetylene Panel Test)	
11	Oxy-Acetylene Panel Test Standardization Organizational Assignments for Studies Conducted October 1961 to	2)
_	February 1962	26
12	General Specifications for Proposed Standard Oxy-Acetylene Panel Ablation Test as of 30 June 1962	
13	Oxy-Acetylene Ablation Test Standardization Working Committee	28
	ILLUSTRATIONS	
Figure	Title	
1	TaC Hot Pressing Study: Effect of Temperature on Density at p = 4500 PSI and t = 30 minutes	
2	Oxy-Acetylene Round Robin Coefficient of Agreement	
3	Heat Flux Studies by Various Laboratories Using Two Different Oxy-Acetylene Burners	
4	Oxy-Acetylene Round Robin Coefficient of Agreement - Mater	
5 6	Oxy-Acetylene Round Robin Coefficient of Agreement - Mater	
b	Oxy-Acetylene Round Robin Coefficient of Agreement - Mater	1al C

### REFERENCES

- (a) NAVWEPS REPORT 7372, "Nozzle and Control Surface Materials for Solid Propellant Rocket Motors," T. O. Anderson, M. Oszust, S. P. Prosen, A. R. Timmins, F. R. Barnet, 1961 (Confidential).
- (b) NAVWEPS REPORT 7390, "The Performance of Selected Plastics Materials in a High Temperature Environment," A. Fisher, M. A. Kinna, F. J. Koubek, F. R. Barnet, 1962.
- (c) NAVWEPS REPORT 7315, "High Temperature Materials Test Facilities at the Naval Ordnance Laboratory," F. J. Koubek, A. R. Timmins, 1961.
- (d) NAVWEPS REPORT 7314, "Ablation Test Methods for Rocket and Heat Shield Materials," F. A. Mihalow, F. J. Koubek, H. A. Perry, 1960.
- (e) NAVORD REPORT 6244, "Experimental Behavior of Reinforced Plastics Surfaces with Hot Gases," H. A. Perry, H. C. Anderson, F. A. Mihalow, 1959.
- (f) Contract NOrd-18136-6, "Refractory Materials Suitable for Use in Guided Missile Propulsion Systems," The Carborundum Company, Quarterly Progress Reports, 1959 to 1962, Confidential.
- (g) Contract NOrd-15536, "Research and Development in Solid-Propellant Rocketry and Related Fields," Quarterly Progress Report No. 46, July Sept. 1961, Atlantic Research Corp., Confidential.
- (h) Contract NOrd-16788, "Improved Insulators for Rocket Motors,"
  Quarterly Progress Report, Nov. 1961 Jan. 1962, U. S. Rubber Co.,
  Confidential.
- (i) "Insulation Development and Sub-Scale Testing," H. Gilbert, Allegany Ballistics Laboratory, Semi-Annual Meeting of S.P.O. Working Group on Refractory and Insulation Materials, 17 18 April, 1962, Confidential.
- (j) "Materials Evaluation," Progress Report Sept. 1961 to June 1962,
   M. J. Heath and R. A. Lott, U. S. Naval Propellant Plant.
- (k) Contract NOrd-15536, "Research and Development in Solid-Propellant Rocketry and Related Fields," Quarterly Progress Report No. 45, April June 1961, Atlantic Research Corporation, Confidential.
- (1) "Evaluation of Rocket Motor Insulation at Atlantic Research Corporation," J. Batchelor, Semi-Annual Meeting of S.P.O. Working Group on Refractory and Insulation Materials, 17 18 April 1962, Confidential.
- (m) "Experimental Evaluation Methods for Ablative Plastics," D. L. Schmidt and H. S. Schwartz, ASD-TR-61-691, March 1962.
- (n) "A Model for the Unsteady Ablation of Charring Thermosetting Plastics," R. D. Mathieu and E. T. Karnowski, M.S.V.D., General Electric Co., July 1962, Confidential.
- (o) "The Thermal Degradation of a Char Forming Plastic During Hypersonic Flight," S. M. Scala and L. M. Gilbert, M.S.V.D., General Electric Co., American Rocket Society Meeting, N.Y., October, 1961.
- (p) "Research on Criteria for Plastic Ablation Materials as Functions of Environmental Parameters," B. McFarland and others,

### REFERENCES

Aerojet-General Corporation, Contract AF33(616)-7401, Progress Reports Aug- Nov. 1960 and Feb-May. 1961.

- Reports Aug- Nov, 1960 and Feb-May, 1961.

  (q) "Aerothermochemistry Studies in Ablation," H. Hurwicz, Avco Corp., Fifth Agard Combustion and Propulsion Colloquium, April 1962.
- (r) "Numerical Analysis of the Transient Response of Advanced Thermal Protection-Systems for Atmospheric Entry," R. T. Swann and C. M. Pittman, N.A.S.A. TN D-1370, July 1962

### INTRODUCTION

- 1. During the past five years, much effort has gone into the development of materials for the inert components of solid fuel rocket propellant systems. In general, the components needed have been thermal insulating liners for the combustion chamber, protective materials for the entrance and exit cones of the nozzle, erosion resistant throat inserts and coatings and substrates for jetevators, etc.
- 2. The high temperature materials program at the Naval Ordnance Laboratory has consisted of a continuing study of materials behavior using a number of laboratory-scale thermal test devices. Information generated in these studies is intended for use as a guide in the improvement of high temperature materials as well as to provide for specific applications. Previous work accomplished at the Naval Ordnance Laboratory in this field is covered in references (a) through (e).
- 3. At the outset of Fiscal Year 1962, there were a number of phases of the program in effect. Tantalum carbide was under investigation for possible optimization as a throat insert material. Commercial nozzle and case insulation materials were being screened on a continuing basis in the oxy-acetylene burner and hydrogen-oxygen rocket engine test facilities. A number of experimental organic insulation systems were under study for possible optimization. The NOL oxy-acetylene panel test method was under study by a working committee, chaired by the Naval Ordnance Laboratory, for possible industry wide standardization. The "alpha-rod" ablation test technique was undergoing further development and refinement, improvements were being made to the plasma arc facility and the arc image furnace was under development as a test facility.
- 4. During Fiscal Year 1962, a number of changes evolved in the above program. Work on refractory type nozzle materials was terminated due to a shift in interest at this Laboratory and because of the progress made by outside contractors in this area. Screening of commercial nozzle and insulation materials was continued on a more selective basis.
- 5. Studies of experimental organic insulation systems was continued on a limited basis due to a manpower shortage. Modified phenolic resins laminated with asbestos mats were the most effective insulation systems studied. Of the modified resins, phenolic-polyamide copolymer resin systems performed the best.
- 6. A thermal insulation study program has evolved using the ablating rod test as the basic tool for study. This work now comprises the main effort of the high temperature materials program at NOL. The general goal of this program is to gain an experimentally supported understanding of ablation-mechanisms with the intention of applying this information to

methods of design and selection of insulation materials as well as to provide a guide in the development and optimization of improved rocket insulation materials. The general plans for this program are to measure the effective ablative properties of materials (such as thermal diffusivity, heat capacity, heat of ablation, etc.) at selected and controlled environmental parameters. Subsequent correlation of properties with the environment would then lead to generalized equations for ablation materials performance and aid in the development and optimization of materials. ablating rod technique known as the NOL "alpha-rod" ablation test was chosen as the method for measuring ablative properties and a plasma arc heater as the environmental simulator. In addition to formulating the plans for the above program and conducting a literature survey of the field, efforts were directed toward the further refinement of the "alpha-rod" test and exploring the requirements for a plasma arc driven, environmental simulator. An arc image furnace was under construction for limited studies using special atmospheres and radiation heating.

- 7. If successful, the above approach to studying ablation phenomena will contribute much to understanding ablating type insulators and aid in the optimization of more efficient systems.
- The Bureau of Naval Weapons working group continued its endeavors to standardize the oxy-acetylene panel test. A round-robin was conducted during the summer of 1961 to evaluate interlaboratory reproducibility of the oxy-acetylene ablation screening test. The study showed poor agreement between laboratories and pointed up the need for further studies of the test method. Detailed studies by the working committee resulted in the adoption of the Victor type 4 No. 7 torch tip as the sole heat source to be used. Another revision was the incorporation of a thin copper disc on the tip of the thermocouple to improve the accuracy and reproducibility of specimen backface temperatures. In addition, a tolerance of ±2% was placed on the accuracy of individual gas flow measurements and copper was substituted for brass on the front face of the transient calorimeter used for measuring heat flux. To evaluate the above revisions a heat flux round-robin was planned with a materials testing round-robin to follow. Satisfactory agreement among laboratories using the revised test method will give the industry a reference test for comparison of ablation type insulation materials produced by various organizations.
- 9. The remainder of this report contains a detailed description of the work accomplished during FY 1962. For the sake of clarity each category is treated separately in its entirety. Nozzle insert materials, insulation materials, insulation studies and oxy-acetylene panel test standardization topics are discussed in order.

### NOZZLE INSERT MATERIALS

### Background

10. During the past several years, the refractory carbides have received considerable attention as candidate materials for nozzle inserts to resist the high temperature erosive effects of metallized, solid propellant exhaust gases. Tantalum carbide, in particular, has displayed varying degrees of success in a number of small scale rocket tests. Preliminary investigations at this Laboratory resulted in a recommendation (ref. (a)) for additional studies of this material.

### Hot Press Studies

- 11. In an effort to optimize the performance of tantalum carbide a program was initiated to study hot-pressing parameters and the effects of various additives on density, grain size, flexural strength and erosion resistance. The initial study consisted of hot-pressing one micron size TaC powder in an inert atmosphere under the following process conditions: temperature, 1700-2650°C; pressure, 2000-6000 psi, and time, 5 to 45 minutes.
- 12. Figure 1 summarizes density data (determined by mercury volumeter method) collected on seven specimens fabricated over a range of temperatures at constant pressure and time. A maximum density of 86.5% of theoretical was obtained at the highest fabrication temperature. The densities obtained were somewhat disappointing in that values exceeding 95% were expected at the high temperature end. Due to the sparsity of data, the reasons for not obtaining higher densities could not be ascertained with any high degree of certainty.
- 13. In the interim, the concentration of NOL effort has shifted almost exclusively to thermal insulation studies. In view of the above and the advances made by other organizations in the refractory carbides field, (ref. (f)), further work by the Naval Ordnance Laboratory in this area is not presently anticipated.

### INSULATION MATERIALS

### General

- 14. During the past year, a number of materials were tested in the oxy-acetylene burner as part of a continuing investigation of experimental materials. Earlier NOL investigations are outlined in reference (b) and cover the period from 1957 to 1961.
- 15. Oxy-acetylene panel test data obtained on typical materials (ref. (b)) are included in Table 1. Materials numbered 1 to 4 are typical rigid and flexible insulation materials presently in use as control specimens in

many bench and small scale rocket tests (refs. (g), (h), (i) and (j)). Material No. 5, a modification of No. 2, produced the highest insulation index of any commercial insulation material tested and material No. 6 produced the lowest erosion rate-highest index combination of any material tested. In general, desirable materials should have erosion rates less than four mils per second, indices of performance less than 20 and insulation indices greater than 180 seconds per inch  $(T_{200} = 45 \text{ seconds})$ .

16. For ease of reporting, the materials tested during the past year have been divided into six categories: modified phenolic resins, rigid insulation materials, flexible insulation materials, silicone rubbers, erosion resistant materials and composite structures. The flame test data are summarized in Tables 2-9 inclusive. A general discussion of these investigations follows.

### Modified Phenolic Resins

- 17. In an effort to improve the thermal insulating effectiveness of phenolic resins, a number of modified phenol-formaldehyde polymers prepared by the Ironsides Resins Co. were studied. In general, four types of modifications were employed: polyamide copolymerization, chlorosilane additions, furfuraldehyde copolymerization and polyamide blending.
- 18. The resins were laminated with asbestos mats (RPD-40) and evaluated in the oxy-acetylene burner. Test results, summarized in Table 2, show that the insulation indices produced were exceptionally high when compared with data obtained from typical insulation materials (Table 1). The phenolic-polyamide resin series were outstanding with the blend type (M-40-N) producing the highest average insulation index number. The phenol-furfural (F-20) and chlorinated phenyl silane (HPCPS-50) series produced lower indices than the polyamide series; however, the former two series did not experience laminate separation at the edges of the specimen that is sometimes characteristic of the latter. Some intumescence of the phenol-furfural specimens was observed during testing.
- 19. From the above study, several observations are readily apparent. Comparison of the test data with those obtained with standard phenolicasbestos mat systems, shows a considerable improvement in insulation effectiveness. Moreover, the polyamide modification appears to be the outstanding resin system. The superior performance of the latter may have been due to its higher volatile matter content and cellular char structure which contribute to increased transpirational cooling. The basic phenol-formaldehyde systems used to produce the modified resins have been obtained from the supplier and tests will be run to provide comparison data for the modified resin systems.
- 20. In view of the excellent torch test performance of these materials and the sparsity of rocket test data on modified phenolics of this type (refs. (j) and (k) show a few tests on commercial grade polyamide-phenolics), four of the resin systems have been selected for evaluation in solid rocket

tests at Atlantic Research Corporation and Allegany Ballistics Laboratory. A phenolic-polyamide copolymer (DP4-43), a phenolic-polyamide blend (M-40-N), a chlorinated phenyl silane (HPCPS-50) and phenol-furfuraldehyde (F-20) will be laminated with asbestos mats early in FY 1963. The resin bath impregnator used for making four-inch specimens has been modified to accommodate eight-inch wide reinforcing cloth or matting. Discs eight inches in diameter, one-half inch thick will be molded and cut up into specimens for solid rocket tests. One disc is sufficient to produce two replicates each for peripheral slab tests at ARC and ABL and the nozzle approach section test at ARC. The use of one molded disc for all three tests is expected to minimize fabrication parameter effects on test results.

- 21. The data in Table 3 were generated to study the effect of phenolic-polyamide resin content on the insulation index. Results tend to indicate that optimum resin content is in the region of 40% (by weight). These results are in general agreement with earlier torch data obtained with conventional phenolic resin systems (ref. (b)). Solid rocket test data on the latter (ref (1)) indicate that in low velocity tests (less than 100 ft/sec.), the insulating effectiveness tends to improve as the resin content is decreased. (Resin contents were studied over a range of 25 to 55%). However, subsequent testing at a higher velocity (1200 ft/sec.) indicated that resin contents in the region of 40-45% performed as well as the lower resin levels.
- 22. Table 4 represents data collected on three new phenolic resin systems laminated with a variety of reinforcing systems. The DP-14 and DP-2 series resins previously exhibited very hard chars when baked at elevated temperatures. The insulation indices produced by the asbestos mat laminates were 267 and 269 secs/inch compared to 229 obtained with a conventional phenolic asbestos mat laminate (Table 1). The nylon-asbestos mat laminate did not perform as well as an all asbestos laminate using the same resin system. It will be noted that the density of the former was not sufficiently low enough to offset the lower insulation index (see II/density values).

### Rigid Insulation Materials

- 23. Oxy-acetylene screening tests on rigid materials supplied by outside organizations are summarized in Table 5. The first of the series, the <u>Thiokol</u> "<u>Ablatalite</u>" materials (a silica microballoon filled epoxy novolak) produced low insulation indices and high erosion rates. While these materials performed poorly, as a monolithic structure, they produced good results when used as a back-up insulation in a composite structure (see Table 9 and para. 35).
- 24. The <u>DuPont "SP" Material</u>, a proprietary molded, unfilled and non-reinforced polymeric material produced results comparable to those obtained with an unfilled, nonreinforced phenolic resin (Table 7). The performance of this material could probably be upgraded by using fillers; however, the effect on the mechanical properties of the material is unknown.

- 25. A number of trowellable, foamed polyester materials, filled with glass forming refractory materials were submitted by the Pittsburgh Plate Glass Co. for evaluation. While the test results were not outstanding, these materials performed quite well for a foamed type structure. The advantages of low density and ease of fabrication could possibly be more fully exploited by imparting higher "char strength" in the system through the addition of phenolic resins and a more refractory filler.
- 26. Samples of <u>Tayloron PA-6</u> (Taylor Corp.) were obtained both as molded material and as a laminate. The test data indicate a reversal in the performance expected of the materials, since the molding compound produced better results than the laminate. Additional specimens are being prepared to prove or disprove the data.
- 27. The low density phenolic-asbestos, MX-5700 materials supplied by the Fiberite Corporation were interesting in that they produced results comparable to a conventional higher density asbestos phenolic molding compound (Table 1). In low velocity solid rocket tests (ref.(1)), the insulating effectiveness of this material was comparable to that obtained with conventional (higher density) asbestos-phenolic insulators. At higher velocities, however, MX-5700 was found to be inferior in performance to the higher density materials.

### Flexible Insulation Materials

28. Oxy-acetylene screening tests on a number of commercial flexible materials are summarized in Table 6. In general, these materials consisted of asbestos filled rubbers and with one exception, there were no improvements in insulation indices over what was already available (see Table 1 - reference data). The exception, V-50 rubber, submitted by General Tire and Rubber Co. produced a high value of 200 secs/inch; however, past experience with abnormally thick specimens would seem to indicate the value to be higher than what would be obtained with a 0.250 thick specimen. The above suspicion is confirmed by low velocity solid rocket tests (ref. (j) and (l)) which showed V-50 to be slightly inferior in performance to V-44 and V-52.

### Silicone Rubbers

29. A number of silicone rubbers, which, as a class, have been previously neglected, insofar as oxy-acetylene screening is concerned, were evaluated. To date, only nonfilled systems have been studied and the data, along with nitrile rubber and phenolic comparison data, are included in Table 7. While none of the silicone rubbers produced outstanding results, it will be noted that, in general, they performed better than unfilled nitrile rubber which is the matrix material for many of the flexible insulators presently in use. Compounding with asbestos or other fillers could possibly produce insulators superior to some of the nitrile rubber-asbestos insulators. Moreover, the low temperature and long term storage capabilities of the silicone rubbers would be an added advantage. In connection with the above, several asbestos filled silicone

rubber composites are being explored and the commercial availability of asbestos filled materials is being surveyed. In connection with the latter, low velocity solid rocket tests on a commercial asbestos filled silicone rubber (Fiberite MX-4730) indicated that the insulating effectiveness was similar in performance to asbestos filled nitrile rubber in one instance (refs. (g) and (l)) and lower (50% higher char rate) in performance at another location (ref. (j)).

### **Erosion Resistant Materials**

- 30. A summary of flame test data on erosion resistant materials is included in Table 8. In evaluating these materials, the emphasis was placed on the erosion rates produced rather than the insulation indices.
- 31. The Samco specimens (Samco No. 7 graphitized fibers bonded with Monsanto SC-1008 phenolic resin) produced scattered results with erosion rates varying from 1.7 to 9.9 mils per second. The use of this low density material might be more fully facilitated if closer control on the molding technique could be obtained.
- 32. The PT-0033, graphitized pitch, specimens supplied by National Carbon Co. produced very low erosion rates which, in combination with the low density, makes it a very interesting material. The data are comparable to that obtained earlier (ref. (b)) with a similar material, PT-0114, which utilizes mascerated graphite cloth in place of the carbon felt.
- 33. The <u>Pluton cloth</u> specimens produced erosion rates that were inferior to that obtained with conventional graphite cloth (Table 1). However, it is interesting to note that the width of the erosion crater formed in these specimens was comparatively narrow and the "burn-through hole" very difficult to detect. Thus, there exists some doubt as to whether "burn-through" was achieved. In view of the above, additional testing is necessary to verify the data obtained.

### Composite Structures

- 34. Data on a number of composite structures submitted for evaluation are summarized in Table 9. While none of the materials surpassed the graphite cloth, asbestos mat reference composite listed at the bottom of the table, a number of observations are worth noting.
- 35. The <u>Vectorite-Ablatalite</u> composite produced results comparable to an asbestos-phenolic molding compound, (Table 1) and showed considerable improvement over the test on Ablatalite used singly (see Table 5).
- 36. The zirconia filled composite showed little or no improvement over what could be obtained with a conventional phenolic asbestos mat laminate. Better results might have been obtained by using a two-ply construction with the zirconia filled phenolic ply on the flame side and the asbestos laminate on the cold side.

### Summary

- 37. Modified phenolics laminated with asbestos mats produced outstanding insulation indices when tested in the oxy-acetylene burner. The most effective system, a phenolic-polyamide blend produced an index number of 402 seconds per inch which is 75% higher than that obtained with a conventional phenolic resin system. Four of the modified resin systems have been selected for solid rocket insulation tests.
- 38. Exploratory screening of silicone rubber indicates that this material, with suitable fillers added, could possibly produce insulation compounds comparable to the nitrile rubber compounds presently in use. Such a compound would be particularly useful for low temperature capabilities and long term storage requirements.
- 39. A proprietary graphitized-pitch bonded carbon felt material exhibited a low erosion rate of 0.9 mils/sec. Low density (0.96 g/cc) combined with low erosion rate make this an interesting class of materials.
- 40. A proprietary, low density phenolic-asbestos material known as MX-5700 produced results comparable to conventional higher density asbestos filled phenolic materials.

### Conclusions

- 41. The above section on insulation materials can be considered to be a fairly representative description of the development efforts of the rocket insulation industry in recent years. In general, these efforts may be summed up as consisting of the modification of existing materials toward optimization for specific areas of application. Some of the better known examples of these efforts are: the orientation of fibrous fillers for maximum insulation effectiveness, the addition of organic and inorganic fillers for increased char strength and transpiration cooling, the flexibilizing of high performance polymeric resins to extend the area of application, the development of techniques for lowering the density of existing systems, etc.
- 42. On a short term basis, particularly in areas of immediate urgency, the above approach has been a fruitful one and a number of insulation systems have achieved a high level of effectiveness for use by the present generation of missiles.
- 43. Over the long run, however, the above approach will undoubtedly lead to a leveling off of productivity as the avenues open for optimization are exhausted and the demands for higher performance systems become greater. Thus, advanced concepts leading to the development of new materials systems capable of more efficient performance are needed.

- 44. In view of the above, and the abundance of effort to maximize the performance of present systems, the Naval Ordnance Laboratory has turned the bulk of its efforts toward the study of insulation materials behavior. Through a better understanding of materials response to various environmental conditions, it is believed that relationships between the environmental parameters and the properties and structure of the material will emerge. With these correlations in hand, the postulation of more advanced systems can begin and more efficient utilization of present systems will be possible.
- 45. A detailed description of the above approach and the progress to date are included in the next section entitled "Insulation Studies."

### INSULATION STUDIES

### General

- 46. During the past several years, the need for better methods in the design and selection of ablating type, insulation materials has been evident. The use of bench and small scale simulators (ref. (m)) has generated voluminous lists of performance data under conditions that are not readily definable and do not simulate any specific application, nor can the data be substituted in any readily available design formulas. At the opposite extreme, theoreticians have developed lengthy equations describing the ablation process, but very little materials properties data are available to substitute in the equations (refs. (n), (o), (p), (q), and (r)).
- 47. During the past year, plans were formulated at the Naval Ordnance Laboratory to pursue a midcourse between bench type screening tests and the highly complicated analytical approach. In general, the program involves utilization of an ablating rod specimen in readily definable environments. Reducing the data obtained into meaningful quantities such as effective thermal diffusivity, conductivity, effective specific heat, heat of ablation, ablation rate, etc.; and correlating these properties with the environmental parameters would then lead to empirical heat transfer equations useful in predicting backface temperatures and in selecting the thickness and type of material needed for a specific application. Moreover, the reverse process could be employed to ascertain the properties needed for various applications and studies conducted to arrive at materials systems possessing these properties.
- 48. A report describing the above program is now being prepared for publication.
- 49. To aid in the planning of the program, a systematic literature search of the field is being conducted on a continuing basis.

### Alpha-Rod Ablation Technique

50. The present technique, known as the NOL "alpha-rod" ablation test (refs. (c), (d) and (e)), is the method that has been selected for measuring the quantities outlined above. This technique consists of continuously measuring the ablation rate and internal temperature of a

moving rod while subjecting it to a controlled heat source. The experimental data are sufficient to calculate the effective thermal diffusivity, heat of ablation and ablation rate. Means for separating thermal conductivity and oc (density - heat capacity product) are now under study.

- 51. During the year, an analog data reduction system for rapid processing of alpha-rod data has been under construction. The first working model is expected to measure the slope of the temperature-time history by means of an optical curve follower, logarithmic converter and electronic differentiator and divide this value into the product of the square of the ablation rate and temperature to give the effective thermal diffusivity. Subsequent modifications are expected to give other properties, such as K and pc as they are developed. By the end of the fiscal year, all but one component of the analog system had been obtained, and calibration, design and construction of auxiliary components were continuing wherever possible.
- 52. The construction of an automatic feed device for the "alpha-rod" test specimen was nearing completion at the end of the year. In brief, this device utilizes an ion sensing probe which measures the "electronic" position of the rod with respect to the flame shield (guard ring) and feeds this information to a servo mechanism, which in turn maintains the face of the ablating rod at a fixed position. Provision is also made for a precision potentiometer to measure the feed rate of the specimen.
- 53. Fabrication of an improved version of the "alpha-rod" flame shield and supporting structure for use with the automatic feed device has been completed. Final assembly and initial trial operation of the automatic feed device is anticipated for the early part of Fiscal Year 1963.

### Variable Environmental Simulator

- 54. To supply the environmental conditions for insulation studies, some form of an electric plasma arc is expected to be suitable for providing a variety of high velocity exhaust gases over a spectrum of pressures and temperatures. When completed, it is expected that this device will be capable of simulating the combustion gas characteristics of present propellant systems as well as those still in the experimental stage.
- 55. In connection with the variable environmental simulator, trips were made to AVCO Corporation, Wilmington, Mass., General Electric Co., MSVD, Valley Forge, Pa., and Johns Hopkins University, Applied Physics Laboratory, Howard County, Maryland, to learn of their plasma are capabilities and methods of studying materials. The equipment which seems to meet the requirements best, at this time, is a plasma are designed by APL. This equipment is said to be capable of operation at high pressures and with low electrode voltages within the capabilities of the present power supply of the NOL water-stabilized arc. It is expected that drawings of the APL are will be supplied at no cost to NOL early in the next fiscal year. In addition to the above, specifications have been submitted to the Plasmadyne Corporation for a cost estimate on a complete facility.

### Arc Image Furnace

- 56. An arc image furnace consisting of two 60-inch mirrors and a carbon arc light source was under construction during the latter half of the year. When completed, this facility will provide a radiation type heat source for material studies in various atmospheres under both static and dynamic conditions.
- 57. Optical equipment for aligning the mirrors of the arc image furnace was received near the end of the year. The receiver mirror has been locked into position and the optical equipment for realignment has been installed with temporary mounts.
- 58. Heat flux reproducibility studies and preliminary materials experiments using the "alpha-rod" configuration are anticipated for the first quarter of the new fiscal year.

### Summary and Conclusions

- 59. An ablation study program has evolved with the general goal to gain an experimentally supported understanding of ablation mechanisms with the intention of applying this information to methods of design and selection of insulation materials, as well as to guide in the development of materials. In general, the program calls for the measurement and correlation of materials properties with the environmental parameters under which they were obtained. The ablating rod technique was chosen as the method for measuring the properties and a plasma arc heater as the environmental simulator. Efforts have been directed toward further refinement of the rod test technique and investigating the requirements for the plasma arc driven environmental simulator. An arc image furnace was under construction for use in radiation heat transfer and special atmosphere studies.
- 60. Upon completion and successful operation of the automatic feed device and data reduction system, it is anticipated that a limited number of insulation materials studies can be conducted using the present high temperature facilities. However, since these facilities are limited in the spectrum of gas chemistry, pressure, velocity, and heat flux needed for a thorough investigation, these studies must be considered exploratory at best, and any extensive efforts must wait for a versatile electric arc driven simulator to provide a wide spectrum of closely controlled parameters. Depending upon the funds available in FY 1963 and the degree of success in obtaining a suitable high pressure plasma arc head design, it is possible that a portion of this facility could be constructed during the year, so that a limited spectrum of environmental parameters could be studied. The power facilities presently used for the water-stabilized arc facility would be used to energize the new facility. A sound suppressor and fume removal enclosure recently constructed for the water arc will house the rocket motor simulator when the latter replaces the water arc.

### OXY-ACETYLENE PANEL TEST STANDARDIZATION

### General

61. During the preceding fiscal year (FY '61) the concept of developing a standard oxy-acetylene ablation test for comparing materials was explored at a meeting called by the Naval Ordnance Laboratory at the request of the Bureau of Naval Weapons (RMMP) and the Special Projects Office (SP-27). The meeting was attended by representatives of various government agencies and private industry. A general test procedure was agreed upon and a "round-robin" test program planned to evaluate the reproducibility of results between laboratories.

### First Round-Robin Test Program

62. The test program was completed early in FY 1962. An analysis of the data, summarized in Table 10 and Figure 2, showed that agreement between laboratories was poor in both the heat flux and the materials tests. The general conclusion reached was that while each laboratory was able to carry out the test within the context of its own techniques and arrive at reasonably reproducible results, there was little or no agreement between laboratories.

### Second Committee Meeting

63. The panel test standardization committee was reconvened at the Naval Ordnance Laboratory on 22 September 1961 to review the round-robin data and formulate plans to improve reproducibility between laboratories. The general feeling was that the variety in commercial equipment used was producing wide variations in test results. The committee recommended that the critical items of equipment and procedure be studied to determine the degree of accuracy and uniformity needed to obtain a standard, reproducible test. Individual committee members volunteered to study such items as flow meter requirements, torch tip characteristics, specimen size and holder design, thermocouple design and mounting, flame pressure and calorimeter measurements, gas purity, etc. The various organizational assignments are summarized in Table 11.

### Third Committee Meeting

- 64. The working committee completed its studies in February, 1962, and its findings were reviewed at a meeting held at the Naval Ordnance Laboratory on 8-9 March 1962.
  - 65. The highlights of the meeting are summarized as follows:
- a. Calorimeter studies revealed that the Victor Type 4 No. 7 torch tip produced a higher heat flux than the Airco No. 10 torch tip

even though port diameters, flow rates, and test distances were the same for both burners (see Fig. 3).

- b. New heat flux data submitted by individual laboratories now show better correlation with 1961 round-robin test data (see Figs. 4,5 and 6).
- c. Reproducibility of heat flux measurements with different calorimeters was found to be satisfactory.
- d. The Victor Type 4, No. 7 torch tip was adopted as standard equipment for the test method.
- e. Tolerances on gas flow rates were established at  $\pm 2\%$  of the specified individual rates and recommendations made to maintain them.
- f. Tolerances on gas purity were adopted to conform with MIL Specs. BB-0925 and BB-Al06.
- g. Specifications for a thermocouple (No. 28 chromelalumel wire) with a five mil thick, 3/16" diameter copper disc (silver soldered to the thermocouple junction) attachment were adopted.
- h. Copper was substituted for brass on the front face of the transient calorimeter to prevent thermal warping.
- i. The need for further consideration of thermal warping experienced with certain classes of test specimens was pointed out. Motions for changing specimen size and configuration were proposed to alleviate this problem but none gained the approval of the committee. Further study of this problem was recommended.
- j. A heat flux round-robin using the standard Victor torch tip was planned. Following successful completion of the heat flux study, a materials round-robin was recommended to evaluate the test method with its latest revisions. It was further recommended that one of the specimens tested be 1/2 inch thick with a 1/4 inch deep thermocouple well incorporated into it to evaluate this method as a means for obtaining more accurate results with specimens that have a tendency to warp.
- 66. The general specifications for the test as it now stands are summarized in Table 12.
- 67. During the remainder of the fiscal year, the members of the working committee were in the process of reequiping their laboratories to conform with the revisions set forth above and in conducting heat flux studies using the revised method. At the end of the year, four of the seven organizations had completed these studies.

- 68. The heat flux round-robin is being coordinated by Raybestos-Manhattan, Inc., Manheim, Pennsylvania. The materials round-robin will be coordinated by the Rock Island Arsenal of the Army Ordnance Corps. The U. S. Rubber Co., Raybestos-Manhattan, Inc., and the General Tire and Rubber Co. will supply test specimens for the program. The materials selected are: USR-3016 and GTR-V-44 (asbestos filled rubbers) and 44-RPD and 41-RPD (asbestos-phenolic laminates). The GTR-V-44 material was selected for the thermocouple well study outlined in paragraph 65. j. above. The target date for delivery of the materials was set for 1 August 1962.
- 69. A listing of the working committee by organization and representatives is included in Table 13.
- 70. Completion of the round-robin test program is expected by January 1963.

### Summary and Conclusions

- 71. The initial round robin evaluation of the oxy-acetylene panel test showed poor agreement between laboratories and pointed out the need for further studies of the test method.
- 72. The ensuing studies resulted in the adoption of the Victor, Type 4, No. 7 torch tip as the sole heat source to be used. A tolerance of  $\pm 2\%$  was placed on the accuracy of individual gas flow measurements and copper was substituted for brass on the front face of the transient heat flux calorimeter. Another revision was the incorporation of a thin copper disc on the tip of the backface thermocouple.
- 73. To evaluate the above revisions, a heat flux round-robin is in progress with a materials round-robin to be completed by 1 January 1963.
- 74. Satisfactory agreement among laboratories will put the committee in a good position to give the industry a suitable reference test for comparison of ablation type insulation materials performance.

### RECOMMENDATIONS

- 75. The screening of commercial nozzle and insulation materials should be continued on the present basis of considering only those materials that appear to hold promise by virtue of data furnished by the supplier.
- 76. The emphasis on the development of advanced insulation concepts through intensive studies of materials behavior should be continued.

- 77. In connection with Item 2, further refinement of the alpha-rod ablation test technique should be pursued. The development of the automatic feed device and data reduction system should be completed and the arc image furnace made operational for rod type tests.
- 78. With the above in hand, exploratory tests should be conducted, in the present high temperature facilities, on representative ablating type, thermal insulation materials.
- 79. Within the limitations of the present test facilities, attempts should be made to correlate the information obtained from the ablating rod tests with the environmental parameters and the structure and properties of the material.
- 80. For maximum effectiveness of the insulation study program, the funding and subsequent procurement of a plasma are driven rocket motor simulator should be actively pursued.
- 81. Upon completion and evaluation of the round robin test program, the oxy-acetylene panel test committee should be reconvened to consider the merits of the test results and to formulate plans for future action.
- 82. On final approval by the committee, the test method should receive formal recognization by the Bureau of Naval Weapons, and other interested activities of the U. S. Government, as the official standard method for laboratory scale screening of ablating type thermal protection materials.
- 83. It is further recommended that the test method, upon finalization, be submitted to A.S.T.M. balloting for consideration as a standard test method.

TABLE 1

OXY-ACETYLENE PANEL TEST<sup>1</sup>/REFERENCE DATA ON TYPICAL MATERIALS

Material No.		Density g/cc	Erosion Rate Density mils/sec g/cc (A)	Time for Back Surface to reach 200°C secs (B)	Index of Performance (A) x 200/(B)	Insulation Index secs/inch (II) (B): thick-	$^{ m II/Density}$
						ness (in)	
ч	Asbestos-phenolic molding compound	1.80	3.6	1.54	16	180	100
ณ	Asbestos mat, phenolic-laminate	1.89	3.6	57.3	13	229	121
₽	PBA <sup>2</sup> /Rubber filled with phenolic and inorganic compounds	1.26	3.5	4.45	12	225	179
4	$ extbf{PBA}^2/ ext{Rubber filled}$ with asbestos	1.29	5.4	53.6	32	136	105
7	Asbestos mat, phenolic-polyamide laminate	1.68	5.5	95.8	7.	383	228
9	Graphite cloth, phenolic laminate	1.503/	1.2	57.0	9	148	8

1/ Standard NOL Test: 225 SCFH gas flow, 1.20  $^{02}/_{\mathbb{C}_{2H_2}}$  (vol.), 0.75 inch test distance, Victor torch, Type 4, No. 7
2/ Polybutadiene-acrylonitrile
3/ Estimated value

TABLE 2

OXY-ACETYLENE PANEL TEST $^{1/}$  DATA PHENOLIC RESIN STUDIES  $^{2/}$ 

Code	Resin 2	Density g/cc	Erosion Rate mils/sec (A)	Time for Back Surface to Reach 200°C secs (B)	Index of Performance (A) x 200/(B)	Insulation Index secs/inch (I_I) (B) thick- ness (in)	I/Density
NPPN	Aklylated phenolic, polyamide copolymer	1.73	2.4	95.0	5	358	207
O4-₩	Phenolic-polyamide blend	1.75	2.2	100.4	<b>.</b> ‡	705 705	230
M-20	Phenolic, polyamide copolymer	1.11	2.5	8.5	9	353	199
TP4-43	Phenolic, polyamide copolymer	1.74	2.3	90.5	50	369	212
F-20	Phenolic-furfural	1.82	3.1	80.4	ထ	<b>ш</b>	171
HPCPS-50	Chlorinated Phenolicsilane copolymer	.c- 1.74	3.1	75.8	ω	291	168
101	Phenolic (control) $\frac{4}{1}$	, 1, 1, 89	3.6	57.3	13	556	121

Standard NOL Test: 225 SCFH gas flow, 1.20 02/C2H2 (vOl.), 0.75 inch test distance, Victor torch-Type 4 No. 7.

Test specimens consisted of resin-asbestos mat laminates, approximately 40% resin (by wt.). data are averages of four replicates Resins supplied by Ironsides Resins, Inc., Columbus, Ohio. જો

Test

resins supplied by ironsides resins, / Ironsides 101 phenolic resin.

TABLE 3

oxy-acetylene panel test $^2$ ) data resin $^2$ / content studies  $^2$ /

Code	Resin Content % (vt.)	Density g/cc	Erosion Rate mils/sec (A)	Time for Back Surface to Reach 200°C Secs. (B)	Index of Performance (A) x 200/ (B)	Insulation Index secs/inch (II) (B) • Thickness (in)	I/Density
DP4-43-1	2	2.04	4.8	33.3	88	149	73
DP4-43-3	01	1.65	2.0	86.3	4	575	526
DP4-43-2	55	1.60	2.3	65.7	9	325	203
DP4-43 <sup>4</sup> /	100	1.23	8.0	29.5	53	113	46

Standard NOL Test (See Table 1 for conditions)
Phenolic, polysmide copolymer, produced by Ironsides Resins, Inc.; Columbus, Ohio
Test specimens consisted of resin-asbestos mat laminates.
Test data are averagesof two replicates.
Single specimen data. नोलोल जो

TABLE 4

LAMINATE REINFORCEMENT STUDIES 2/ OXY-ACETYLENE PANEL TEST<sup>2</sup>/DATA

							Insulation	
		Resin Content		Erosion Rate	Time for Back	Index of Performance	Index secs/inch (Ir)	
Code	Reinforcement	% (wt.)	Density g/cc	Density mils/sec g/cc (A)	ਧ _	(A) x 200/ (B)	(B) thickness (in)	$^{ m I}_{ m I/Density}$
DP14-19-A	Asbestos Mat	ድ	1.63	2.5	67.2	7	592	165
DP14-19-02/	Glass Cloth	33	1.96	5.4	32.1	ネ	128	65
DP14-31-G <sup>4</sup> /	Glass Cloth	ネ	2.04	4.4	8.64	18	193	95
DP2-16-A	Asbestos Mat	<del>1</del> 11	1.69	3.3	9.99	10	267	158
DP2-16-R	High Silica Glass Cloth	8	1.79	8.4	24.0	04	8	55
DP4-435/	Asbestos Mat	04	1.74	2.3	90.5	5	<b>69</b> £	212
/Zzn-nan 19	Nylon-Asbestos Mat	04	1.44	2.9	9.69	6	253	176
DP4-436/	None	100	1.23	8.0	29.5	53	11.3	ਲ ਲ
								. <b>⊃~</b> .

Standard NOL Test (See Table 1 for conditions).
Using modified phenolic resins produced by Ironsides Resins, Inc., Columbus, Ohio.
Test data are averages of two replicates.
Molding pressure: 200 psi.
Molding pressure: 1,000 psi.
Average of 4 replicates.
Single specimen data. नोला निमानी

TABLE 5

OXY-ACETYLENE PANEL TEST<sup>1</sup>/DATA

_
TYPE
(RIGID
MATERIALS
INSULATION
COMMERCIAL

Description of Specimens	Density g/cc	Erosion Rate mils/sec (A)	Time for Back Surface to Reach 200°C secs (B)	Index of Performance (A) x 200/(B)	Insulation Index secs/inch (II) (B) thickness (in)	II/Density
Ablatalite 2A $\frac{2}{}$	0.77	7.6	15.9	122	19	87
Ablatalite $2^{3}$	0.73	14.7	12.9	228	611	29
DuPont "SP" Material $2/$	1.43	5.9	37.8	32	146	102
Refractory filled foamed polyester material No. $68 ^{\frac{1}{2}}$	1.17	11.3	22.8	96	8	77
Refractory filled foamed polyester material No. 69 $\frac{\mu}{4}$	1.29	8.5	30.7	55	112	87
Asbestos-phenolic laminate PA-6 $\frac{5}{2}$	1.70	4.1	38.0	22	159	ま
Asbestos-phenolic molding compound PA-6 $\frac{5}{2}$	1.74	3.0	68.5	σ	281	162
Asbestos-phenolic molding compound $MX-5700^{\frac{1}{2}}$	1.27	3.6	7.94	16	204	161
Low Density MX-5700 $\frac{1}{4}$	1.11	5.9	T. 44	18	191	172

Standard NOL Test (see Table 1 for conditions). Silica microballoon filled epoxy novalak; Thiokol-Reaction Motors, Denville, N.J.; average of two replicates

E. I. du Pont de Nemours, Wilmington, Del.; average of two replicates.
Pittsburgh Plate Glass Co., Pittsburgh, Pa.; No. 68, aver. of three replicates; No. 69, aver. of four replicates.
Taylor Corp., Valley Forge, Pa., one replicate only.
Fiberite Corporation, Winona, Minn., molding pressure 500 ps1, average of two replicates.

/ Molding pressure 200 psi.

TABLE 6

OXY-ACETYLENE PANEL TEST<sup>1</sup> DATA

COMMERCIAL INSULATION MATERIALS (FLEXIBLE TYPE)

		Erosion	Time for Back Surface to	Index of	Insulation Index	
Description of Specimens	Density g/cc	mils/sec (A)	Reach 200°C secs (B)	Performance (A) $\times$ 200/(B)	secs/inch (II) B : thickness (in)	<sup>1</sup> I/Density
Asbestos-Buna S Rubber 2	1.81	9.9	34.1	39	135	75
Asbestos-Neoprene Rubber 2/	1.80	9.9	29.5	45	9118	99
Asbestog-Buna N Rubber, $V-44$	1.29	2.0	15.8 5/	55	150	911
Asbestog-Buna N Rubber, $V-50 \frac{1}{2}$	1.29	5.3	/9 L.69	6	500	155
Asbestos-Buna N Rubber, V-52 $\frac{11}{4}$	1.34	0.9	33.0	35	<b>†</b> 11	85
Asbestos-phenolic filled rubber molding compound, LS-10107 7	1.2	4.8	33.3	20	711	8
Felt-phenolic filled rubber laminate, LS-10108 $\overline{I}/$	1.35	ट. १	42.6	&	170	126

75% asbestos; Raybestos-Manhattan, Inc., Bridgeport, Conn.; aver. of four replicates. 70% asbestos; Raybestos-Manhattan, Inc., Bridgeport, Conn.; average of four replicates. Standard NOL Test (see Table 1 for conditions)

General Thre and Rubber Co., Akron, Ohio; average of two replicates Abnormally thick specimen, 0.705.

Abnormally thick specimen, 0.749.

Raybestos-Manhattan, Inc., Manheim, Pa.

TABLE 7

OXY-ACETYLENE PANEL TEST<sup>1</sup> DATA SILICONE RUBBERS

				Time for		Insulation	
	Description of	Density	Erosion Bate	Back Surface to Reach 200°C	Index of Performance	$ m secs/inch(I_I)$	IT/
	Specimen	g/cc		secs (B)	$(A) \times 200/(B)$	(1n.)	-/ Density
	Silastic S-2048 $\frac{2}{}$	1.33	11.2	20.0	112	82	62
	Silestic $s-6510^{2}$	1.24	15.8	15.9	199	61	64
	RTV-60 2/	1.38	38.7	3.3	2350	19	7,
	RTV-88 2/	1.43	20.2	15.5	261	94	32
	<b>se-</b> 555 <b>2</b> /	1.28	6.2	28.9	45	103	&
	Buna N $\frac{1}{4}$	1.00	25.3	10.0	506	04	0 <del>1</del>
၁၁	Phenolic $\frac{14}{2}$	1.25	6.2	57.0	玄	148	118
							•

1/ Standard NOL Test (See Table 1 for conditions).
2/ Dow Corning Corp., Midland, Mich., Average of two replicates.
3/ General Electric Co., Waterford, N.Y., average of four replicates.
4/ Typical data for non-reinforced materials.

TABLE 8

OXY-ACETYLENE PANEL TEST DATA

COMMERCIAL EROSION RESISTANT MATERIALS

		Erosion			Insulation Index	
Description of Specimens	Density g/cc	Kate mils/sec (A)	to Reach 200°c secs (B)	Index of Performance (A) x 200/(B)	<pre>secs/inch (I<sub>I</sub>) (B) + thickness (in)</pre>	I/Density
Samco No. 7 Carbonaceous fibers, phenolic resin binder 2/	.73	6.7	7.0	191	<b>%</b>	%
Carbon felt and graphitized pitch binder, PT-0033	%.	6.0	1.5	120	9	9
Pluton "A" carbon- ized cloth laminated with phenolic resin \( \frac{1}{4} \)	. ed 1.40	9.8	23.3	48	93	<b>3</b> 8.
Pluton "B-10" carbonized cloth laminated with phenolic resin \frac{\psi}{}/	1.27	8.4	19.4	87	7.7	61

Standard NOL test (see Table 1 for conditions). નોળો

Space Age Materials Corp., Woodside, N. Y.; average of three replicates; resin binder - Monsanto SC-1008

National Carbon Co., Cleveland, Ohio; average of three replicates.

NOL fabricated; cloth supplied by Minnesota Mining and Mfg. Co., St. Paul, Minn.

resin binder - Ironsides 101, 40% resin content; average of two replicates.

TELM

TABLE 9

# OXY-ACETYLENE PANEL TEST<sup>±/</sup> DATA

# COMPOSITE STRUCTURES

Description of Specimens	Density 8/cc	Erosion Rate Density mil/sec g/cc (A)	Time for Back Surface to Reach 200°C secs (B)	Index of Performance (A) x 200/(B)	Insulation Index secs/inch (II) (B) thickness (in)	I/Density
Vectorite-Ablatalite composite 2/	ı	3.5	0.94	15	185	1
Asbestos Mat, zirconia, phenolic composite $\frac{1}{2}$	1.85	5.6	52.3	10	211	411
Asbestos Mat, phenolic laminate $\frac{1}{4}/$	1.62	3.2	54.8	12	217	134
Sanco No. 8, carbonaceous fibers, asbestos, phenolic composite $\frac{5}{2}$	0.95	6.2	22.0	26	72	76
Graphite cloth, asbestos mat, phenolic laminate $6/$	ı	2.3	74.5	9	292	1

Ablatalite (a silica microballoon filled epoxy novolac); Thiokol-Reaction Motors, Denville, N.J.; Front face - Vectorite laminate of alternate layers of graphite and glass cloths; backface -Standard NOL Test (see Table 1 for conditions) one replicate only. न्गेला

content 17.2% (by weight). Raybestos-Manhattan, Inc., Bridgeport, Conn.; average of two replicates. Similar to the outer plies mentioned in 3/ above, but 0.250 thick. Front face, approximately 3/16" of phenolic bonded Samco No. 8 fibers; backface 1/16" asbestos Two outer plies, each 0"100 thick, asbestos mat (45%), phenolic (55% wt.) laminate. Inner ply,  $\sim$ 

sheet; Space Age Materials Corp., Woodside, N.Y.; aver. of three replicates; Monsanto SC-1008 

NOL fabricated; front face-graphite cloth, phenolic-polyamide resin laminate, 0:150 thick, backface-Graphite asbestos mat, phenolic polyamide resin laminate, 0"105 thick; average of two replicates. Graphicloth - WCB grade, National Carbon Co; asbestos mat, RPD-40, Raybestos-Manhattan, Inc; phenolicnylon resin, SF-4057, Reichhold Chem. Co. phenolic resin. े

Table 10 AMALYSES OF ROUND-ROBIN DATA (OXX-ACETYLENE PANEL TEST)

1	MOL		8	Ravbestos 1		Victor Torch		Group	Rock	Island			Goodrich			ABC		Atre	Airco Torch Group Gen. Tire	anough a	Þ	3. Rubber	ş
Material A	m	ပ		A	o	Ą	1 1	ы	Ą	æ	ပ	Y	m	0	1	a	ပ	۲	_	υ	H		٥
7 99	ŭ	Density 1.866 1.120 1.231 1.827 1.105 1.218	1.827	1.105	1.218	1.876	1.024	1.217	1.809	1.104	1.218	1.752	1.108	1.220	1.877	1.104	1.219	1.815	1.108	1.222	1.831	1.140	1.198
. 0035 .	.0810 8 .0054	. 6463 . 1300.	26. 8 27.80 7.00 7.00	. 4562 8 . 4750	.070. 8 57.20.	.0321 6 .0081	.0825 7 .0115	.0661 5 .0334	.0029 .	.07522 . 8 .00240 .	.0298 .0050	4520. 8 0053	84.00. 8 1000.	.0022	2656 8 1710	0900 8 2010:	.0327 8 0030	988 889	2465 8 1400.	.02.54 5 .000.7 5	24.8 8 8 8 8 8	ος Θ	88 88 803.5 15
	- 0803 8 8 50052 9	οθο. 13	• •		.020. 8 8 1.0103	.0164 .0052 8	00,000	.040. 20059		•	.0072 8 .0046 17	98.00. 98.00. 99.00.	1 ½ 8.00 1 ½ 8.00 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	, 200. 8 8	848 95 %	.0833 1910. 151	, 88 80. 88 828	12.00 17.00 12.00 12.00	, 20. 8 8 8 8 8	, 4210. 1000. 1	2810. 8 4200.	.0018 8000.	. gʻ∞ <b>g</b> ʻ⊐
αo -≠	. 02148 . 0750 . 8 8	.0386. 8 1200.	.01710. 8 .0126 74	.0584 .0165 7 8 .0051 .0043 9 26	.0165 .0043 .0043		.0135 17 17	.039 <b>1</b> 5 .0067 17				.0075 8 5000.	.0536 8 11	44420. 8 8 9 9 9	វិត <sub>∞</sub> ថ្នុំជ	2180 8 2110 41	.0018 0.0018	0101 8 0100. 16	45. 8. 8. 8. 8. 8. 8.	124 1000 1			
9. 88.∞ <del>9</del> . ₩	. 0778 . 0050 6	.0373 8 8 19	.0105 8 .0035 33	.0563 .0048 8	.0157 8 4400.	. 0800 20020 20020	0167 7 10123	.0379 5 .0073 1.9				.0069 7	.0526 8 .0056	.0241 8 4200. 10	2110. 8 0100. 9	0108 14	.0052 8 .0017 7						
0114 8 0038 33	.0569 8 8 9 9 9	. 000 900 18	.00. 80. 80. 80. 80. 80.	.0472 8 120.11	9410. 8 7400.	.0094 .0020 .21	.0666 8 8 14	.0377 8 .0062 .16	90 8 8 8 8 8	0628 . 8 0106 . 17	0251 8 .0036 14	.000. 8 4000.	.0500. 8 .0056	.0240 .0023 10	0110 8 0100. 9	889. 8 600.	9 0100.	.000.000.000.000.000.000.000.000.000.0	.0037 8 8 8	9220 6 5 5 5 5	8 8 8 9 a	.0033 8 8 8	ogio. 8 gioo. 9
			This in	This set not used in analysis.	bead.																		
84 2 64 4	netrat: replicative signative signature signat	]/ Average penetration rate of 100°C thermal front, $\frac{2}{2}$ /Number of replicates. $\frac{7}{2}$ / Standard deviation. $\frac{4}{1}$ / $S_1\beta^*=S_1/p_R\times 100$ . $\frac{5}{2}$ / Average erosion rate, cm/sec	of 100'	c them	ual fron	it, cm/sec.	· 0																

### TABLE 11

### OXY-ACETYLENE PANEL TEST STANDARDIZATION ORGANIZATIONAL ASSIGNMENTS FOR STUDIES CONDUCTED OCTOBER 1961 TO FEBRUARY 1962

Organization	Assignment
Atlantic Research Corporation (W. Wyatt)	Furnish design drawings and specifications for a thermocouple probe assembly, a specimen holder and a photoelectric detector for determining burn-through.
General Tire and Rubber Co. (W. Hartz)	Study heat flux characteristics of selected commercial torch tips. Explore the feasibility of obtaining design drawings for a custom made torch tip.
General Tire and Rubber Co. (W. Hartz)	Study the effect of specimen size and thickness on test results.
B. F. Goodrich Co. (E. Rowe) (D. Silvey)	Investigate flow meter and pressure gage requirements needed for accuracy and reproducibility of flow-conditions.
U. S. Rubber Co. (R. Keller)	Study thermocouple junctions and thermocouple disc sizes.
Rock Island Arsenal (R. Ofner)	Comparison of Pressure Probe Data with NOL's data.
Raybestos-Manhattan, Inc. (J. Wronski)	Calorimeter Study - Raybestos, Goodrich and U. S. Rubber (Interchange of 3 calorimeters)
U. S. Naval Ordnance Laboratory (W. McLean)	Furnish design drawings for NOL flame pressure probe.
U. S. Naval Ordnance Laboratory (D. Caum)	Obtain information on purity data and requirements for oxygen and acetylene.

### TABLE 12

GENERAL SPECIFICATIONS FOR PROPOSED STANDARD OXY-ACETYLENE PANEL ABLATION TEST AS OF 30 JUNE 1962

Torch Tip - Victor, Type 4, No. 7 with water cooling jacket provided by tester.

Total gas flow rate - 225 SCFH

Ratio of oxygen to acetylene - 1.20 (volume)

Tolerance on individual gas flow rates - ±2%

Gas purity - To conform with Mil Specs BB-0925 and BB-Al06

Testing distance - 0.750 ± 0.015 inches

Angle of impingement - 90 ± 3°

Thermocouple - No. 28 chromel-alumel wire with 3/16 inch diameter copper disc, 5 mils thick silver soldered to junction.

Spring loaded to maintain contact with backface of specimen.

Specimen - 0.250 ± 0.015 inches thick. No limitations\* on length and width (or diameter in the case of circular specimens).

\* Four inch x four inch and four inch diameter specimens have been found to be adequate for the purpose.

TABLE 13

## OXY-ACETYLENE ABLATION TEST STANDARDIZATION WORKING COMMITTEE

Organization	Address	Representative
*Rock Island Arsenal	Rock Island, Illinois	R. Ofner
*U. S. Naval Ordnance Lab.	Silver Spring, Maryland	F. J. Koubek**
Ordnance Materials Research Office	Watertown, Massachusetts	M. P. Marks
Bureau of Naval Weapons	Washington, D. C.	S. J. Matesky
*General Tire and Rubber Co.	Akron, Ohio	W. Hartz
*U. S. Rubber Company	Naugatuck, Connecticut	D. Beretta
*B. F. Goodrich Research Center	Brecksville, Ohio	D. H. Silvey
*Thickol Chemical Corp.	Denville, N.J.	R. Keller
*Raybestos-Manhattan, Inc.	Manheim, Pa.	J. Wronski
Narmco, Inc.	San Diego, Calif.	J. Chin
Aerojet General Corp.	Sacramento, Calif.	A. A. Stenersen
Atlantic Research Corp.	Alexandria, Va.	J. B. Murphy
Allegany Bállistics Lab.	Cumberland, Maryland	C. Rousseau
Prewitt Plastics Company	Alexandria, Va.	W. B. Wyatt

<sup>\*</sup> Organizations participating in the round-robin program. \*\* Committee Chairman

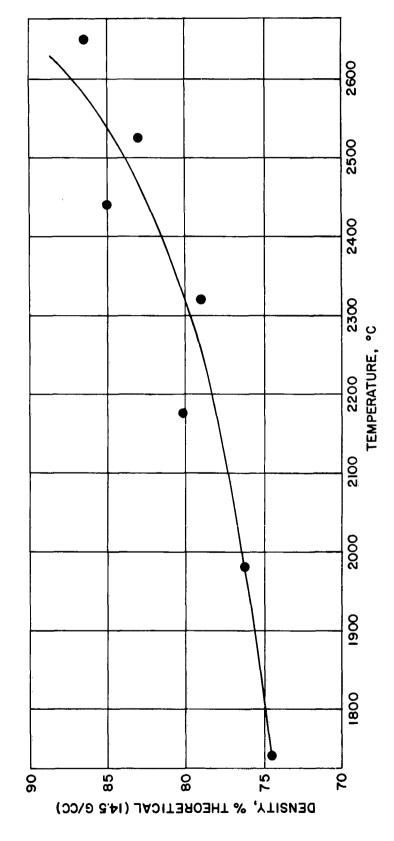
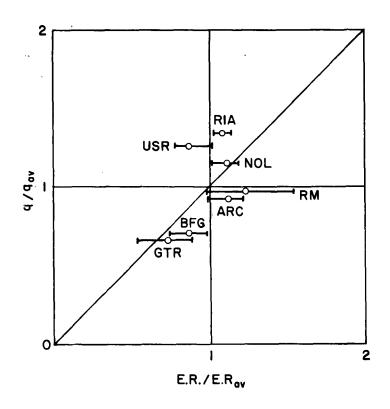


FIG. I Tac HOT PRESSING STUDY: EFFECT OF TEMPERATURE ON DENSITY AT P = 4500 PSI AND 1 = 30 MINUTES



USR-U.S. RUBBER CO.
RIA - ROCK ISLAND ARSENAL

NOL- NAVAL ORDNANCE LABORATORY

RM - RAYBESTOS-MANHATTAN, INC. ARC-ATLANTIC RESEARCH CORP.

ARC - ATLANTIC RESEARCH CORP. BFG - B. F. GOODRICH CO.

GTR-GENERAL TIRE AND RUBBER CO.

q ≠INDIVIDUAL LABORATORY HEAT FLUX DATA

q<sub>av</sub> = AVERAGE HEAT FLUX DATA OF ALL LABORATORIES

E.R. = EROSION RATES FOR INDIVIDUAL LABORATORIES

E.R.<sub>av</sub> AVERAGE EROSION RATES FOR ALL LABORATORIES

THREE MATERIALS TESTED

= AVERAGE OF THREE
 MATERIALS TESTED

FIG. 2 OXY-ACETYLENE ROUND ROBIN COEFFICIENT OF AGREEMENT

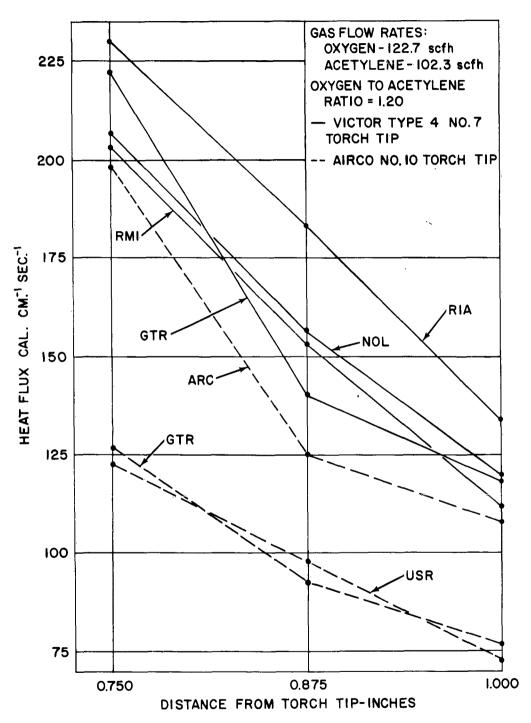


FIG. 3 HEAT FLUX STUDIES BY VARIOUS LABORATORIES USING TWO DIFFERENT OXY-ACETYLENE BURNERS

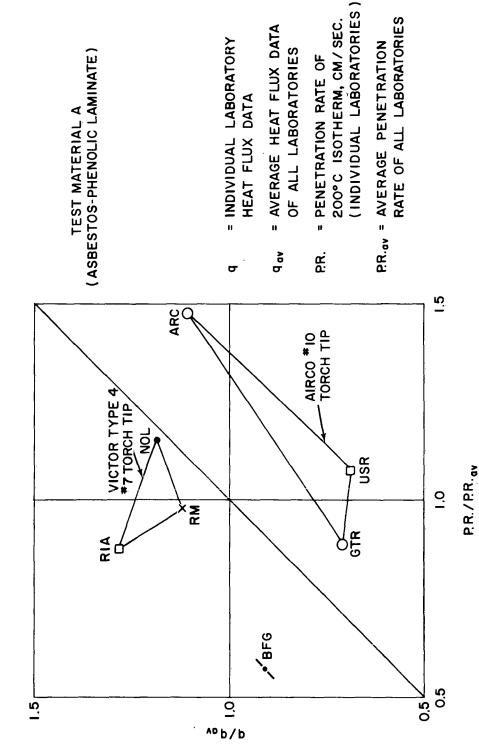


FIG. 4 OXY-ACETYLENE ROUND ROBIN COEFFICIENT OF AGREEMENT

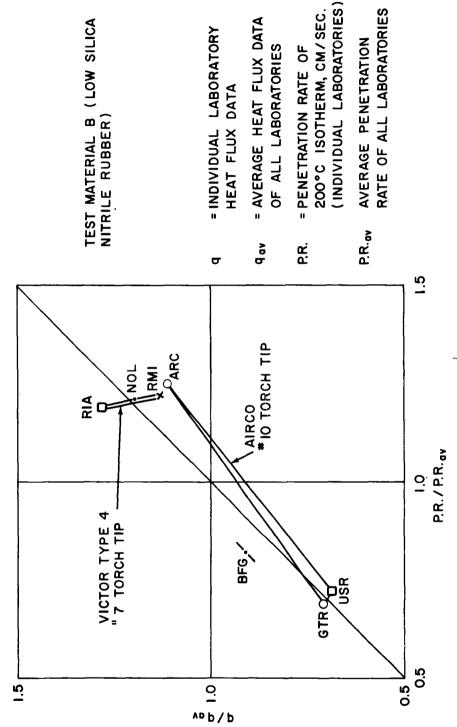


FIG. 5 OXY-ACETYLENE ROUND ROBIN COEFFICIÊNT OF AGREEMENT

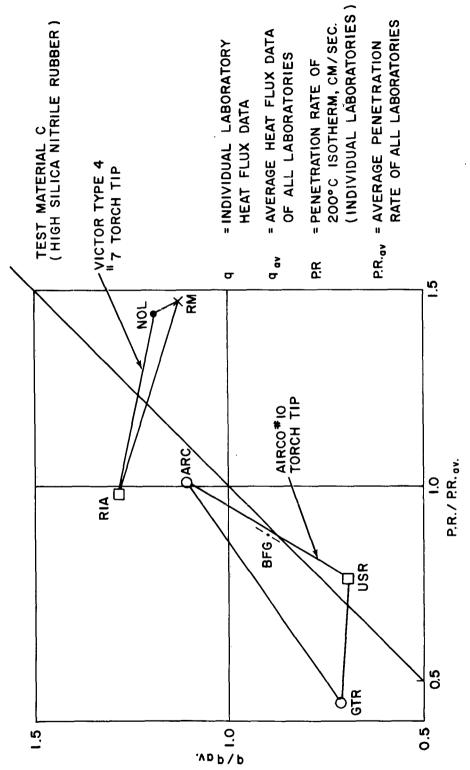


FIG. 6 OXY-ACETYLENE ROUND ROBIN COEFFICIENT OF AGREEMENT

#### DISTRIBUTION

Copies BUWEPS, RMMP-11 (I. Silver) RMMP-23 (S. Matesky) RRMA-35 (J. Gurtowski) RRMA-2 (T. F. Kearns) RMGA-811 (J. Wright) 4 DIS-3 Director, Special Projects Office Department of the Navy Washington 25, D. C. Attn: SP-20 SP-2714 (H. Bernstein) Bureau of Naval Weapons Representative Lockheed Missile Systems Division P. O. Box 504 Sunnyvale, California Allegany Ballistics Laboratory P. O. Box 210 Cumberland, Maryland 4 Attn: Library Atlantic Research Corporation Shirley Highway and Edsal Road Alexandria, Virginia Attn: G. Olcott Aerojet-General Corporation Sacramento, California Attn: A. Ginsburgh W. O. Wetmore 6 Bureau of Naval Weapons Resident Representative Aerojet-General Corporation P. O. Box 1947 Sacramento, California Attn: SPIA 30 Aerojet-General Corporation P. O. Box 296 Azusa, California Attn: Librarian Georgia Institute of Technology Engineering Experiment Station Atlanta, Georgia

Attn: J. D. Walton

	DISTRIBUTION	Copies
Chemical Propulsion Information Applied Physics Laboratory The Johns Hopkins University 8621 Georgia Avenue Silver Spring, Maryland	on Agency	3
Ordnance Materials Research Of Watertown Arsenal Watertown 72, Massachusetts Attn: ORDBE-Z	ffice	
Office of Naval Research Department of the Navy Washington 25, D. C. Attn: Code 423, Metallurgy	Branch	
U. S. Rubber Company Research and Development Depar Wayne, New Jersey Attn: Dr. P. Viohl	rtment	
Commander Air Force Ballistic Missile Da Air Force Unit Post Office Los Angeles 45, California Attn: WDAT	ivision	
Jet Propulsion Laboratory 4800 Oak Grove Drive Pasadena 3, California Attn: I. E. Newlan Chief, Reports Group		
Thickol Chemical Corporation Elkton Division Elkton, Maryland Attn: Librarian		
Thickol Chemical Corporation Utah Division Brigham City, Utah Attn: Library Section		
Thickol Chemical Corporation		

Redstone Division

Huntsville, Alabama Attn: Library Section

......

#### DISTRIBUTION

Copies
Commander
Armed Services Technical Information Agency 10
Arlington Hall Station
Arlington 12, Virginia
Attn: TIPDR

Department of the Army Office, Chief of Ordnance Washington 25, D. C. Attn: ORDIB

Commanding Officer Picatinny Arsenal Dover, New Jersey Attn: Library

Commander

Army Rocket and Guided Missile Agency Redstone Arsenal, Alabama Attn: Technical Library ORDXB-OTL

Commander
Army Ballistics Missile Agency
Redstone Arsenal, Alabama
Attn: ORDAB-ESI

Commanding General
Ordnance Ammunition Command
Joliet, Illinois
Attn: ORDLY-AREL, Engr. Library

Commanding Officer
Harry Diamond Laboratory
Washington 25, D. C.
Attn: ORDTL (012)

Commander, U. S. Naval Ordnance Test Station China Lake, California Attn: Code 4526 Code 5557

Armour Research Foundation of
Illinois Institute of Technology
Technology Center
Chicago 16, Illinois
Attn: M. J. Klein, Chemistry Division

3

2

### DISTRIBUTION

Copies

Commanding Officer
Rock Island Arsenal
Rock Island, Illinois
Attn: ORDBC-9320

American Machine and Foundry Company Mechanics Research Department 7501 N. Natchez Ave. Niles 48, Illinois Attn: A. D. Kafadar

Bjorksten Research Laboratories, Inc. P. O. Box 265 Madison 1, Wisconsin Attn: Librarian

Hercules Powder Company Bacchus Works Magna, Utah Attn: Librarian

Battelle Memorial Institute Ceramics Division 505 King Avenue Columbus 1, Ohio Attn: J. Lynch

Defense Metals Information Center Battelle Memorial Institute 505 King Avenue Columbus 1, Ohio Attn: C. S. Dumont

Air Force Flight Test Center Edwards Air Force Base, California Attn: FTR SC/2nd Lt. L. B. Thomas

Commander
Aeronautical Research Laboratories
Wright-Patterson Air Force Base, Ohio
Attn: RRLC

Commanding Officer
Naval Propellant Plant
Indian Head, Maryland
Attn: Technical Library

#### DISTRIBUTION

Copies

Minnesota Mining and Manufacturing Co. Central Research Department 2301 Hudson Road St. Paul 9, Minnesota Attn: Library

Union Carbide Research Institute P. O. Box 324 Tuxedo, New York Attn: Dr. Verner Schomaker

Commander

Commander
Mare Island Naval Shipyard
Vallejo, California
Attn: Code 372, Rubber Laboratory

Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio
Attn: Mr. D. L. Schmidt (ASRCNC-2), Non-Metallic Materials Div.
Mr. R. E. Hendrick (ASRCNE-1), Non-Metallic Materials Div.

DISTRIBUTION	Copies
Naugatuck Chemical Division U. S. Rubber Company Naugatuck, Connecticut Attn: R. McCoy	oogacs
National Aeronautics and Space Administration 1510 H Street N.W. Washington 25, D. C. Attn: RSM	2
Commander Air Force Ballistic Missile Division Air Force Unit P.O. Los Angeles 45, California Attn: WDAT	
Grand Central Rocket Company P. O. Box 111 Redlands, California Attn: Helen Ashman, Librarian	
National Academy of Sciences Materials Advisory Board 2101 Constitution Avenue, N.W. Washington 25, D. C. Attn: Panel on Rocket Nozzle Materials	
B. F. Goodrich Aviation Products P. O. Box 395 Rialto, California Attn: A. B. Japs, Manager Rocket Motor Development	
Armed Services Technical Information Agency Arlington Hall Arlington, Virginia	10

CATALOGING INFORMATION FOR LIBRARY USE

			E	BLIOGRAPHIC	BIBLIOGRAPHIC INFORMATION			
	DESCRI	DESCRIPTORS		copies			DESCRIPTORS	cobes
SOURCE	NOL technical report	ort.		NOLTR	SECURITY CLASSIFICATION AND CODE COUNT	ICATION	Unclassified - 36	96 <b>ø</b> n
REPORT NUMBER	63-3			636663	CIRCULATION LIMITATION	TATION		
REPORT DATE	30 Jan. 1963	,		Ø163	CIRCULATION LIMITATION OR BIBILIOGRAPHIC	LATION		
					BIBLIOGRAPHIC (SUPPL., VOL., ETC.)	73		:
			S	UBJECT ANAL	SUBJECT ANALYSIS OF REPORT			
	DESCRIPTORS	CODES		DESCRIPTORS		CODES	DESCRIPTORS	CODES
High te	High temperature	нтем	Ablation			ABLA	Furnace	FURW
Resistant	$\operatorname{nt}$	REST	Thermal			THER	Radiation	RADT
Materiels	ટી	MATE	Insulati on	ជ		INSL	Atmospheric	ATMO
Missile		MISL	Alpha			AL PH	Materials (Testing)	MATET
Nozzles		NOZZ	Rod			RODS	Materials (Design)	MATED
Temperature	ture	TEMP	Test			TEST	Materials (Study)	MATES
0xy		OXYZ	Plasma		,	PLAM	Polyamide	POLI
Acetylene	ne	ACEY	Arc			ARCS	Co-polymer	COFO
Torch		TORC	Driven			DRIV	Silicone	SILN
Phenolic	v	PHEN	Environmental	ntal		ENVI	Rubber	RUBB
Resins		RESI	Simulator	_		SIML	Elastomeric	ELAT
Comme <b>rc</b> ial	ial	CMMR	Image			TMAG	Insulator	INSA

Naval Ordnance Laboratory, White Oak, Md.  (NOL technical report 63-3)  HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS (U), by F. J.  Koubek, 30 Jan, 1963, 28p, diagr., tables. Tasks RAMP 23-054/212-1/F009-06-06-03 and NOL 554.  Oxy-acetylene torch tests on phenolic resture sins are described, also those conducted on a 4. Rocket motors Internal insulation study program is described. Intending to apply information to improved methods of design and selection of insulators. Efforts have been directed toward refinement of "alpha-rod" test technique and investigat- ing requirements for plasma are driven en- vironmental simulator. An are image furnace tis under construction for radiation and spe- unolassified.	Mayal Ordnance Laboratory, White Oak, Md.  (NOL technical report 63-3)  HIGH TEMPERATURE RESISTANT MATERIALS FOR  MISSILE PROPULSION SYSTEMS (U), by F. J.  Koubek, 30 Jan, 1963. 28p, diagr., tables.  Tasks RAMP 23-054/212-1/F009-06-003 and  NOL 554.  (NOLLASSIFED  OXY-acetylene torch tests on phanolic resists are described, also those conducted on a ture  sins are described, also those conducted on a ture  Naterials  Thermal insulation study program is described.  Thermal insulation study program is described.  Intending to apply information to improved methods of design and selection of insulators  of "alpha-rod" test technique and investigat—III. Project ing requirements for plasma are driven en-  vironmental simulator. An are image furnace is under construction for radiation and spe-
Mayal Ordnance Laboratory, White Oak, Md.  (NOL technical report 63-3) HIGH TEMPERATURE RESISTANT MATERIALS FOR MISSILE PROPULSION SYSTEMS (U), by F. J. Koubek, 30 Jan. 1963. 28p. diagr., tables. Tasks MAMP 23-054/212-1/F009-06-003 and NOL 554.  Oxy-acetylene torch tests on phenolic resilunt to manber of commercial ablation type materials. Thermal insulation study program is described. I. Title into apply information to improved in Noubek, methods of design and selection of insulators.  Efforts have been directed toward refinement of "alpha-rod" test technique and investigat. III. Project ing requirements for plasma are driven en-vironmental simulator. An arc image furnace is unclassified.	Naval Ordnance Laboratory, White Oak, Md.  (NOL technical report 63-3)  HIGH TEMPERATURE RESISTANT MATERIALS FOR Materials - MISSILE PROPULSION SYSTEMS (U) by F. J.  Koubek. 30 Jan. 1963. 28p. dlagr., tables. Tasks RAMP 23-054/212-1/7009-06-003 and UNCLASSIFIED  OXy-acetylene torch tests on phenolic resistance of commercial ablation type materials. Thermal insulation study program is described. Title intending to apply information to improved methods of design and selection of insulators.  Efforts have been directed toward refinement of "alpha-rod" test technique and investigat. III. Francis J., title ing requirements for plasma are driven en- vironmental simulator. An are image furnace is under construction for radiation and spe- dial atmosphere studies.